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# TAPE CASTING AS AN APPROACH TO AN ALL-CERAMIC TURBINE SHROUD SEAL

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## SUMMARY

Gas paths seals have a one-dimensional variation in material requirement. Tape casting is a method which allows the fabrication of thin ceramic sheets, which may be laminated to accommodate these requirements. Using tape casting, thin sheets of zirconia (0.25 mm) were fabricated. These castings were successfully laminated and fired without bloating or delamination, demonstrating the feasibility of this approach.

## INTRODUCTION

Efforts directed toward the improvement of the turbine tip seal strive for improved gas turbine engine performance (ref. 1). This may take the form of either decreased specific fuel consumption, increased maximum thrust, or increased component service life. The substantial economic benefit is obtained through a reduction of specific fuel consumption. This may be effected by reducing the clearance between the turbine blade and the seal, increasing engine operating temperatures, decreasing cooling air requirements, and decreasing component weight.

This places several requirements on the turbine shroud seal. The surface of the seal exposed to the blade must be abrasible so that in the event of a rub, blade wear is avoided. At the same time it is necessary for the surface to survive the impact of particulate matter, both ingested and produced within the engine, with a minimum of erosion. The hot face must also be of a refractory nature, able to withstand high temperatures without melting or subliming. The point of attachment must be made from a material capable of providing structural support. Finally the entire seal must be able to withstand the thermal gradients and thermal cycles which occur in the engine.

The major variation in seal requirements occurs in the radial direction, or in other words in the layers "parallel" to the seal surface (see the schematic of fig. 1).

An approach which has had success in the use of a superalloy casting onto which layers of other materials are plasma sprayed (ref. 2). Both layers of yttria stabilized zirconia (YSZ) and YSZ - NiCrAlY mixtures are used. In this way it is possible to change materials, and hence behavior, to accommodate the spatially varying requirements such as those outlined previously. Some problems exist with this system, however, thermal strains arise due to the thermal expansion mismatch between metal and YSZ, oxidation of the metal phase occurs under the ceramic with an accompanying volume change, and it is difficult to

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optimize the plasma sprayed YSZ material between abrasability and erosion resistance.

By comparison the fabrication of the entire seal from zirconia offers several advantages. These include the elimination of any oxidation since zirconia is already an oxide, a reduction in cooling air requirements since the thermal conductivity of zirconia is much lower than that of the metal, a reduction in thermal strains since there are no discontinuities in the material, and the use of a single material permits greater control and freedom in the fabrication procedure.

The mechanical properties of zirconia are dependent on the chemical composition (impurities present), the heat treatment history, and the distribution of the pore phase. Pure zirconia exhibits a disruptive phase transformation at 1170 °C (2130 °F) which prevents its use as a refractory material (ref. 3). This transformation may be avoided through the incorporation of cation impurities, such as Mg, Ca, or Y, which stabilize the high temperature crystallographic form. The best engineering properties have resulted from partially-stabilized material which contains a mixture of the polymorphs cubic, tetragonal, and monoclinic. Both magnesium-partially stabilized zirconia (ref. 4), Mg-PSZ, and calcia-partially stabilized zirconia (ref. 5), Ca-PSZ, when properly fabricated lead to material with high strength (up to 600 MPa, 87 ksi) and high fracture toughness (up to 6.0 MN/m<sup>3/2</sup>, 6.4 ksi √in) through a process termed transformation toughening. This material is suitable for the structural back side of the seal. An additional, and notable, property of transformation toughened material is that abrasion of the surface produces an increase in bend strength (ref. 6). The important implication of this is that the production of surface flaws through finish machining or relative motion between the seal support rails and the engine case should not weaken the material.

Neither Mg-PSZ or Ca-PSZ are suitable for use near the hot face of the seal for two reasons. First, the mechanical properties degrade when the materials are exposed to temperatures above 900 °C (1650 °F) for long periods of time and secondly, both calcium and magnesium are subject to preferential evaporation leaving unstabilized zirconia. Yttria-partially stabilized zirconia (between 5 and 12 cation mol %) on the other hand, does not display transformation toughening and therefore does not have the structural qualities of Mg-PSZ and Ca-PSZ. It does, however, have better thermal cycling behavior as a result. A second quality of this material is that the stabilizer, yttrium, does not preferentially evaporate. This material is, therefore, well suited for use on the hot face while not appropriate for use at the rails.

Thus, analogous to the metal/ceramic designs, in an all-zirconia seal it is desired to produce a laminated structure. In the all-zirconia seal, the goal is to produce discrete layers of differing chemistry arranged in a controlled manner, which includes curvature of each layer. A method of achieving this is to use tape casting to form thin flexible sheet, laminate the sheets by pressing, and then sinter to form a monolith.

Tape casting is an established method for producing thin ceramics (ref. 7). This involves placing the powder of the desired chemistry into suspension. The suspension is spread onto a hot surface, the liquid is evaporated and a thin sheet of green ceramic is left. The cast is given flexibility through the incorporation of polymers (called plasticizers) dissolved in the

suspending liquid. Since the sheets are fabricated independently, the chemistry of any one is independent of the others. To construct an actual seal, these thin sheets are stacked in the appropriate order, Y-PSZ for the hot face region and Ca-PSZ for the structural region, and compressed into a monolith under pressure. An advantage of powder technology is that a burnout phase may be included in the suspension which will decompose to leave pores during the sintering heat treatment. Through the selection of burnout phase, control over the size and shape distribution of the pores as well as their number density may be exerted. This allows freedom in the optimization of the erosion/abrasion behavior for the top layers. In order to demonstrate the feasibility of this approach a set of preliminary experiments were carried out. The key points to be demonstrated were that lamination and firing could be accomplished without bloating or delamination. Bloating can occur when a material contains organic which decompose and produce gaseous products. If pathways do not exist to vent these gases substantial pressures can buildup and the ceramic deforms.

### EXPERIMENTAL PROCEDURE

These experiments were done using only one material, Ca-PSZ, and no attempt to vary the porosity or chemical composition was made.

A laboratory scale tape casting machine, based on that described in reference 8, was constructed, see figure 2. The casting surface was a 15 by 30 cm (6 by 12 in) piece of window glass, heated by six 100 W light bulbs. Their intensity was controlled by a conventional dimmer switch. The blade for spreading the suspension was made of teflon and mounted on a micrometer head for height adjustment.

The tape casting procedure used was based on one developed for the tape casting of alumina (ref. 7). This procedure was not optimized and should be regarded as preliminary. As outlined in table I, the milling was a two-step process. An alumina mill was charged with the zirconia powder,<sup>1</sup> the suspension media, and a deflocculant. The mill was run overnight then the plasticizers and binder were added and the mill was run overnight a second time. The suspension was de-aired and cast onto the glass surface which was at a temperature between 38 and 40 °C (100 and 104 °F). The glass surface was lubricated with a mixture of soybean oil and lecithin dissolved in ethyl alcohol (PAM, General Foods). The casts were removed from the glass with a metal spatula. Typical casts were on the order of 10 cm, (4 in) in mean diameter and could be removed as a single piece.

Discs of 18 mm (0.75 in) were punched from the casts. To form laminates, 10 to 30 of these discs were stacked in a hardened steel cylindrical die and compressed under 140 MN/m<sup>2</sup> (20 ksi) at room temperature. In some cases a drop of solvent was placed in the middle of the stack, however it was found that this was not necessary for the stack to become bound.

Two different sintering environments were used. In the first case the atmosphere was air and the heating rate of 450 °C/hr (842 °F/hr) was used. The

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<sup>1</sup>Zircoa Corning Solon OH, 3.4 wt % CaCl<sub>2</sub>.

soak temperature was 1600 °C (2910 °F) and the soak time was ~4 hr.<sup>2</sup> The second employed a vacuum furnace capable of higher temperatures. The total pressure in the furnace was less than 10<sup>-6</sup> torr. In this case a slower heating rate of 100 °C/hr (212 °F/hr) was used with a soak temperature of 1800 °C (3270 °F) and a soak time of 6 hr. After sintering in a vacuum the samples were given a 10 min anneal in air at 1400 °C (2550 °F) in order to restore stoichiometry.

## RESULTS AND DISCUSSION

### Casting and Lamination

Using the procedure outlined in table I castings were produced, with an average thickness near 0.25 mm (0.01 in). The cast thickness was increased by more than a factor of five when the glass surface was roughened by spraying it with a fluorocarbon die lubricant. Release of this thicker cast was, however, considerably more difficult.

It was found that if the glass temperature was much greater than 40 °C (104 °F), boiling of the liquid occurred resulting in the entrapment of bubbles. In addition, an effort to eliminate toluene was made, due to its corrosive effect on the seal around the lid of the ball mill. However when additional ethyl alcohol was substituted for toluene, a skin formed on the surface of the suspension and subsequent bubbling beneath the skin occurred. Drying times, normally a few minutes, also increased dramatically when the skinning occurred.

Pressing of the single casts into a laminate was routinely accomplished at 140 MN/m<sup>2</sup> (20 ksi). Sheets of paper were generally placed on both ends of the stack to facilitate release after pressing. A drop of solvent was sometimes included in the middle of the stack prior to pressing. This was found to be unnecessary and excessive solvent softened the plasticizers allowing appreciable flow out of the die cavity past the rams.

### Firing

Two sets of single layers were fired in air to 1600 °C (2910 °F). One set was simply laid on a piece of plasma sprayed Y-PSZ, the second set was placed between two pieces of plasma sprayed YPSZ, each weighing less than a gram. The results are shown in figure 3. The free standing samples suffered extensive warpage while that under the light load remained flat and had a radial shrinkage roughly 15 percent. Some shrinkage cracks appear around the periphery of the flat sample, however these could likely be avoided through modification of the firing schedule.

The sintering of laminates made up of 30 discs was carried out in both an air atmosphere to 1600 °C (2910 °F) and under vacuum to 1800 °C (3270 °F). The results were markedly different in the two cases and are shown in figure 4.

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<sup>2</sup>Some estimation of the time is involved since a power failure occurred during the high temperature soak.

The sample fired in air showed no visual evidence of bloating, cracking, or delamination while sample fired in the vacuum furnace showed extensive bloating and some delamination, despite its slower heating rate. The precise cause of the difference in behavior is unknown. Bloating occurs when the material becomes impervious prior to the completion of the decomposition of binders and plasticizers which produces gaseous species. There are two factors which may have contributed the pronounced bloating in the vacuum fired material. First, the lack of oxygen may have retarded the decomposition of the polymers to require a higher temperature and longer time which allows greater densification of the ceramic to take place prior to completion of the gaseous evolution. Secondly the stoichiometry of the zirconia was changing during the sintering heat treatment. This flux of oxygen through the material may have enhanced the rate of densification in the near surface region.

By contrast, the air fired sample appeared very satisfactory. No detectable delamination occurred. Some slight cracking near the edge was observed, however this appeared to correlate well with flaws introduced by the paper liners in the die during pressing. As in the case of the single casts roughly 15 percent radial shrinkage was observed. No warpage of free standing samples was observed.

Though these experiments are preliminary, this sample is a demonstration of the fact that a relatively thick ceramic piece, 5 mm (0.2 in), can be made from a laminate of relatively thin, 0.2 mm (0.01 in), green ceramic sheets. What needs to be addressed in the future is the sintering behavior of a laminate in which the type of stabilizer varies between the layers and the procedure for designing in porosity. Optimization of the tape casting procedure and sintering schedule requires further work.

## CONCLUSIONS

(1) The fabrication of a ceramic monolith through the sintering of a laminated green body made from thin sheets is feasible. Such a method allows discrete changes in chemistry to be effected across the monolith.

(2) Tape casting and pressing is a viable method for the fabrication of such a laminate.

(3) The sintering of a ceramic body containing a high fraction of decomposable polymers was accomplished without bloating when fired in air, however, severe bloating occurred when sintering took place in a vacuum.

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TABLE I. - TAPE CASTING PROCEDURE

Step 1 *	Charge mill with	1000 gm zirconia w/Ca stabilizer
		415 gm toluene
		160 gm ethyl alcohol
		20 gm corn oil (deflocculant)
Step 2 *	Mill overnight	
Step 3 *	Add to mill	180 gm daiso prepolymer type A
		215 gm polyglycol E-800
		200 gm Butvar B98
Step 4 *	Mill overnight	
Step 5 *	Cast onto glass at	38-40 C



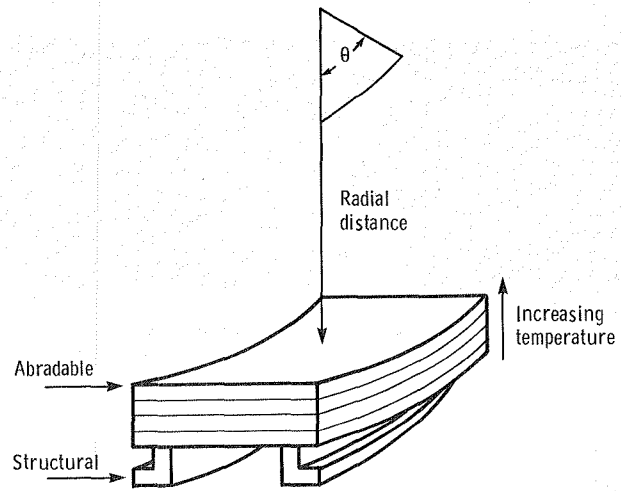


Figure 1. - Schematic of turbine tip seal indicating the seal requirements at different positions of radial distance.

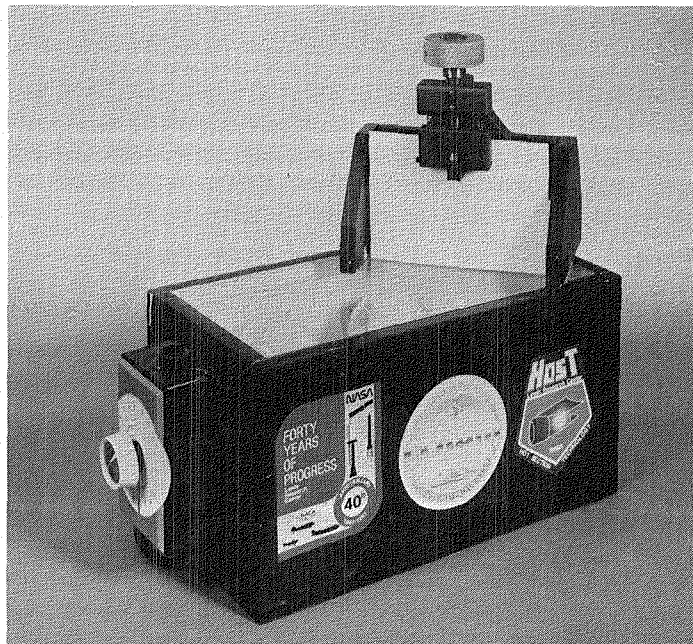


Figure 2. - Photograph of the laboratory scale tape casting machine.

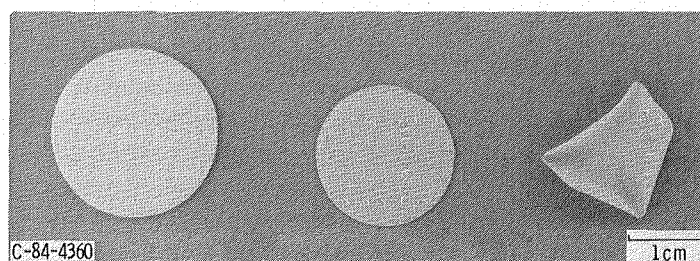


Figure 3. - Photograph of single castings.

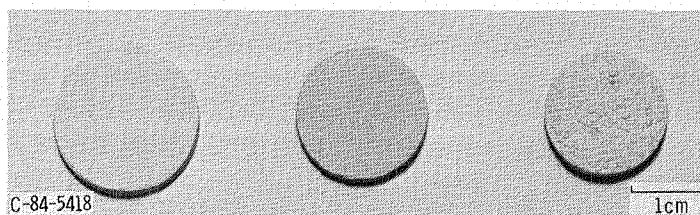


Figure 4. -Photograph of laminates of thirty single castings.

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